

1/5

Solvent	Dilution (D) and Washing (W)	Yield(%)	N <sub>9</sub> – N <sub>7</sub> Isomers	Temp	NaOH
DMF	D: Toluene W: Toluene/Acetone	96%	N <sub>9</sub> -(79%); N <sub>7</sub> -(18%)	160°C	+
DMF	D: Toluene W: Toluene/Acetone	93%	N <sub>9</sub> -(77%); N <sub>7</sub> -(19%)	150°C	+
DMF	D: Toluene W: Toluene/Acetone, crystallize	70%	N <sub>9</sub> -(97%); N <sub>7</sub> -(1.5%) Crystals from EtOH	160°C	+
DMF	D: Toluene W: Toluene/EtOH(cold)	67%	N <sub>9</sub> -(91%); N <sub>7</sub> -(3.3%)	150°C	+
DMF	D: Toluene W: Toluene/Acetone, crystallize	56%	N <sub>9</sub> -(98%); N <sub>7</sub> -(1.56%) Crystals from EtOH	130°C	NaOEt
DMF	D: Toluene W: Toluene/EtOH(cold)	95%	N <sub>9</sub> -(55%); N <sub>7</sub> -(12%) Un-reacted starting	160°C	-
DMF	D: Toluene W: Toluene/EtOH(cold)	70%	N <sub>9</sub> -(83%); N <sub>7</sub> -(6%)	150°C	-
DEF	D: Toluene W: Toluene/EtOH(cold)	89%	N <sub>9</sub> -(77%); N <sub>7</sub> -(21%)	150°C	+
DMA	D: Toluene W: Toluene/EtOH(cold)	70%	N <sub>9</sub> -(85%); N <sub>7</sub> -(6%)	150°C	+
DMA	D: Toluene W: Toluene/EtOH(cold)	84%	N <sub>9</sub> -(73%); N <sub>7</sub> -(19.5%)	150°C	-

Table 1

Figure 1

2/5

Solvent	Dilution (D) and Washing (W)	Yield(%)	N <sub>9</sub> – N <sub>7</sub> Isomers	Temp	NaOH
DMA	D: None, evaporated to dryness and t-BME added W: t-BME	100%	N <sub>9</sub> -(76%); N <sub>7</sub> -(20%)	150 <sup>0</sup> C	+
DMA	D: None, evaporated to dryness and CH <sub>3</sub> CN added W: CH <sub>3</sub> CN	92%	N <sub>9</sub> -(80%); N <sub>7</sub> -(16%)	150 <sup>0</sup> C	+
DMA	D: None, evaporated to dryness and EtOH added W: EtOH	67%	N <sub>9</sub> -(90%); N <sub>7</sub> -(4%)	150 <sup>0</sup> C	+
DMA	D: None, evaporated to dryness and IPA added W: IPA	84%	N <sub>9</sub> -(92%); N <sub>7</sub> -(3%)	150 <sup>0</sup> C	+
DMA	D: None, evaporated to dryness and IPA/CH <sub>3</sub> CN (1:1) added W: IPA/CH <sub>3</sub> CN (1:1)	91%	N <sub>9</sub> -(95.25%); N <sub>7</sub> -(2.49%)	150 <sup>0</sup> C	+
DMA	D: Evaporated to half in volume and Toluene added W: Toluene/ EtOH(cold)	87%	N <sub>9</sub> -(76%); N <sub>7</sub> -(12%)	160 <sup>0</sup> C	+
DMA	D: Evaporated to half in volume and Toluene added W: Toluene/ EtOH(cold)	89%	N <sub>9</sub> -(68%); N <sub>7</sub> -(15%)	140 <sup>0</sup> C	+

Table 2

Figure 2

3/5

Solvent	Dilution (D) and Washing (W)	Yield(%)	N <sub>9</sub> – N <sub>7</sub> Isomers	Temp	Cat
DMA	D: CH <sub>3</sub> CN W: CH <sub>3</sub> CN	94%	N <sub>9</sub> -(88%); N <sub>7</sub> -(11%)	150 <sup>0</sup> C	+
DMA	D: EtOAc W: EtOAc	89%	N <sub>9</sub> -(90%); N <sub>7</sub> -(8%)	150 <sup>0</sup> C	+
DMA	D: Toluene W: Toluene/EtOH (cold)	95%	N <sub>9</sub> -(92%); N <sub>7</sub> -(7.5%)	150 <sup>0</sup> C	+
DMA	D: Toluene W: IPA (cold)	97%	N <sub>9</sub> -(86%); N <sub>7</sub> -(13%)	150 <sup>0</sup> C	+
DMA	D: IPA W: IPA (cold)	91%	N <sub>9</sub> -(97%); N <sub>7</sub> -(1.34%)	150 <sup>0</sup> C	+
DMA	D: IPA W: IPA	87%	N <sub>9</sub> -(97%); N <sub>7</sub> -(1.15%)	160 <sup>0</sup> C	+
DMA	D: IPA W: IPA	82%	N <sub>9</sub> -(98%); N <sub>7</sub> -(0.96%)	140 <sup>0</sup> C	+
DMA	D: t-BME W: IPA	87%	N <sub>9</sub> -(95.4%); N <sub>7</sub> -(2.78%)	150 <sup>0</sup> C	+
DMF	D: Toluene W: Toluene/EtOH (cold)	93%	N <sub>9</sub> -(91%); N <sub>7</sub> -(3%)	Reflux	+

Table 3

Figure 3